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Chelation ion chromatography as a technique for trace elemental analysis in complex matrix samples

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Abstract

A new method for the determination of lanthanides based on chelation and ion chromatography with absorbance detection is described. γ-Aminobutyrohydroxamate resin or its derivative, *N*-methyl-γ-aminobutyrohydroxamate resin in a column was used to concentrate and separate lanthanides from the alkali and alkaline earth metals and other matrix components. Various complexing agents were investigated as possible eluants. The influence of a sample matrix and parameters important for quantitative analysis are discussed. Using γ-aminobutyrohydroxamate resin as concentrator, and potassium nitrate (0.02 M) as matrix eliminator, with the mobile phase of oxalic acid (0.03 M, pH 4.5)–diglycolic acid (0.005 M), and 4(2-pyridylazo)resorcinol (6×10^{-5} M)–ZnEDTA(1×10^{-4} M) as postcolumn reagent, most of the lanthanides can be separated and determined in the complex matrix samples. The detection limit was found to be 2.5 ng ml^{-1} for these elements by concentrating 25 ml of sea water.

Keywords: Chromatography; Chelation ion chromatography; γ-Aminobutyrohydroxamate resin; Preconcentration; *N*-Methyl-γ-aminobutyrohydroxamate resin; Lanthanide separation

1. Introduction

Interest in rapid and efficient separation of the lanthanide elements has recently increased, because ultrapurified rare earth metals show unique electronic, optical and magnetic properties. Hence, research on the separation and purification of rare earth metals has been carried out intensively for producing new advanced materials. However, to obtain high purity products, the separation of adjacent elements in the rare earth series is very energy-demanding. A conventional solvent extraction is considered to be the best method for the treatment of a large amount of rare earth ore, but a great deal of solvent and extractant are required to obtain high purity products. Thus a new, simpler method to enhance the separation efficiency is desirable [1].

The determination of trace elements in complex matrices remains one of the most challenging areas of analytical chemistry. Complex matrices include sea water, brines, estuarine waters, as well as biological, botanical and geological materials. In general, these matrices have high levels of alkali and alkaline metals, with trace levels of the transition elements.

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High levels of these metals cause significant interferences and sensitivity losses for most analytical techniques used for trace metal determination. Metal ions could be separated on a stationary phase, made of resin or silica material, on which a suitable ligand is immobilized. One of the most common chelating stationary phases is Chelex-100, which exhibits different complexing abilities towards various transition metals. The retention of the cations is controlled by the concentration of the complexing agent and the pH of the eluent. Currently, chelation ion chromatography (CIC) [2–10] is considered a very powerful technique for trace and ultra trace ion analysis in complex matrices. The benefit of coupling chelation and ion chromatography provides a technique that makes possible the determination of trace elements in complex matrices that have proven to be difficult or impossible to analyze by ion chromatography or conventional atomic spectrometry. This technique was first reported by Kingston et al. [2]. They used the imminodiacetate chelating columns synthesized by Dionex and not the commercial Chelex-100 for the determination of trace transition and rare earth elements. They claimed that Chelex-100 with low cross-link (gel-type) microporous polystyrene-divinylbenzene (PS-DVB) supporting polymer would result in a low recovery of metal ions due to the physical degradation of the resin under pressure (>100 psi). They synthesized a highly crosslinked macroporous PS-DVB containing the imminodiacetate functional group which is the same as that of Chelex-100. Recently, high-performance chelation ion chromatography has been developed and extensively studied by Jones et al. [3,7–9]. They used the chelating-dye impregnated resin for the determination of trace metals in calcium-containing matrices [7], coastal sea water [8] and offshore oil-well brines [9].

In previous work in this laboratory, γ-aminobutyrohydroxamate resin and its derivatives have been synthesized and their coordination behavior toward transition elements determined by EPR spectroscopy, electronic spectrscopy and potentiometry [11–13]. This report focuses on the preconcentration and separation of lanthanides based on the γ-aminobutyrohydroxamate resin as well as its *N*-methyl derivative, and on-line connection of γ-aminobutyrohydroxamate resin and Dionex CS-5 columns.

2. Experimental

2.1. Apparatus

The ion chromatography system (Model 4000i, Dionex, Sunnyvale, CA) which includes a gradient pump and UV–visible spectrophotometer was used for the applications. All data were recorded by a model SP datajet integrator (Spectra-Physics, Santa Clara, CA). A column packed with *N*-methyl-γ-aminobutyrohydroxamate resin (*N*-methyl P13) (230–325 mesh, 8% cross linking) prepared as previously [12,13] or an IonPac CS-5 column (Dionex) was used for the separation of lanthanides. The dimension of the separator is 25 cm length with 3 mm i.d. The chromatographic system was assembled as in Scheme 1, in which two metal-free valves were used. One served to convey the eliminating solution or cleaning solution, the other was for the sample solution. A switching valve was installed to control the sequence of solutions in the system. Pretreatment prior to separations was achieved on a home made guard and concentrator column. γ-Aminobutyrohydroxamate resin (P13) (100–230 mesh, 8% cross linking) was used for both the guard column and the concentrator column (50×2 mm i.d.). Here the concentrator was used for simultaneous preconcentration and matrix elimination.

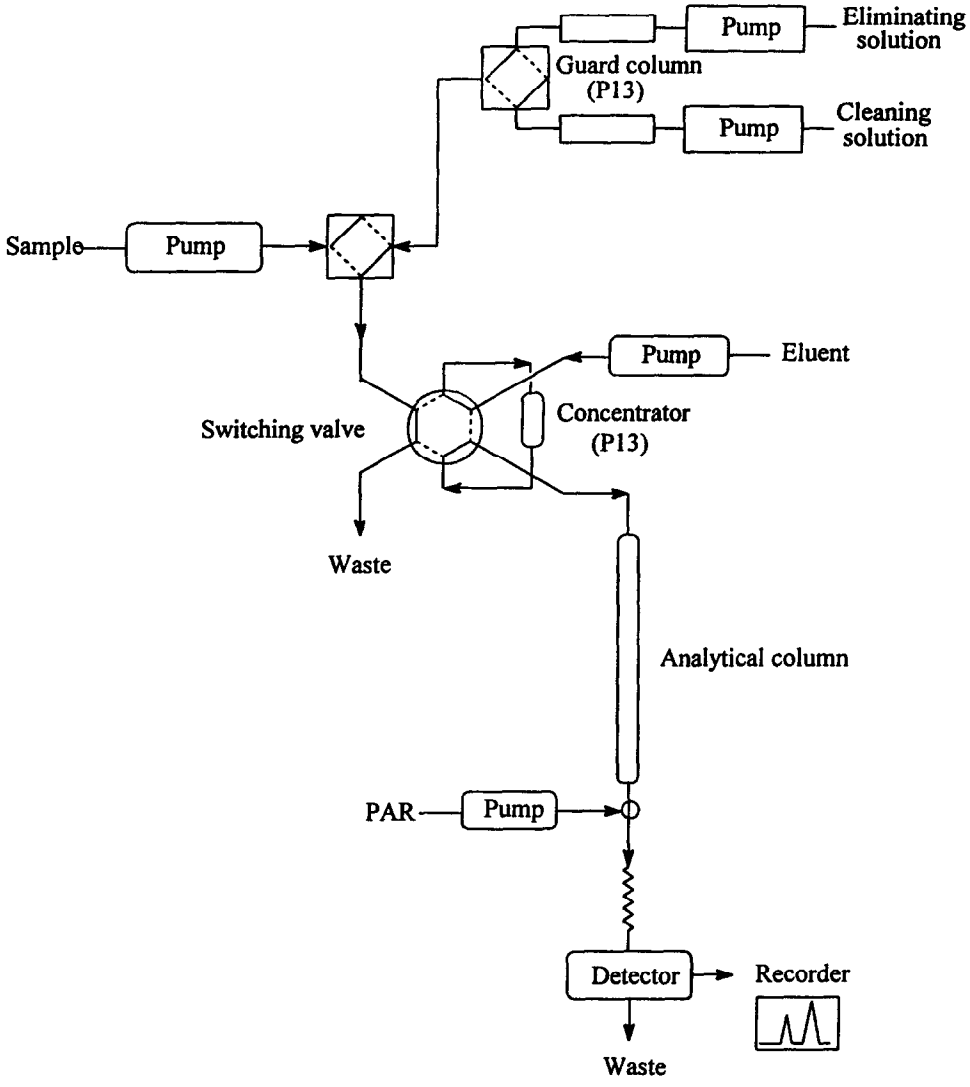
A pH meter (Radiometer PHM 61, Copenhagen) was used to measure the pH of the solutions.

2.2. Synthesis of the chelating resins

The detailed procedures for the preparation of γ-aminobutyrohydroxamate resin (P13) [11,12] and *N*-methyl-γ-aminobutyrohydroxamate resin (*N*-methyl P13) [12,13] have already been reported. In this paper, only the flow chart is given (Scheme 2). The metal capacity of these resins toward the lanthanides is 1.5–1.9 mmol g^{-1}.

2.3. Reagents

All eluents and standards were prepared from analytical reagent grade (Merck, Darmstadt) and pure water (Milli-Q Ultra Pure Water System, Millipore) under a class 100 laminar flow hood. Stock solutions

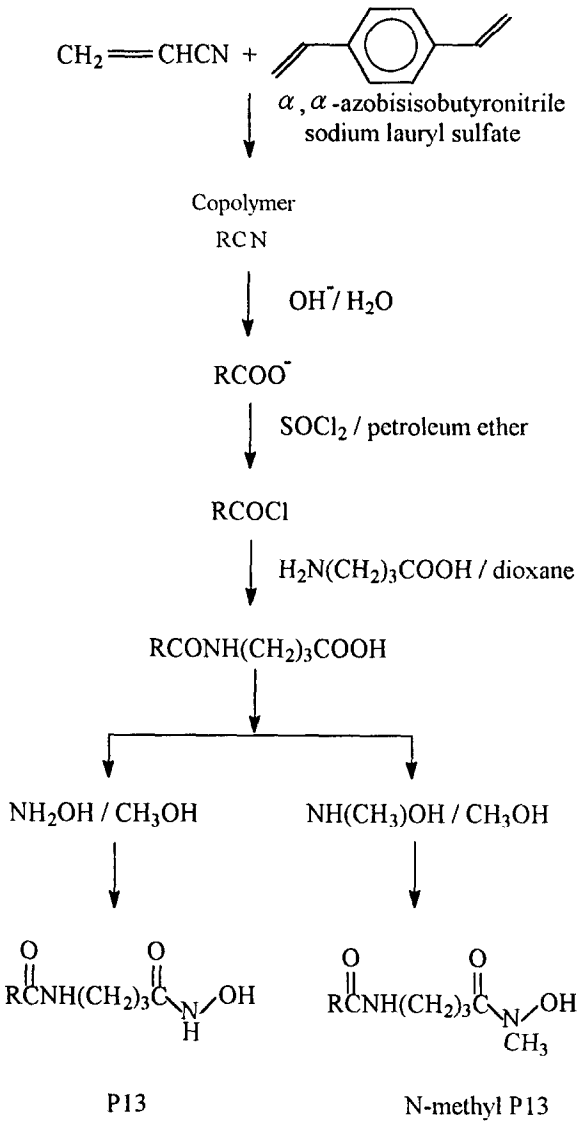


Scheme 1. Schematic diagram for the on-line preconcentration system.

of lanthanide chlorides (1×10^{-2} M) were prepared by dissolving weighed amounts of the oxides (99.9%, Aldrich) in 2.0 M hydrochloric acid, evaporating carefully to a viscous residue followed by dissolution in pure water. The solutions were diluted to the desired volume and standardized by titration with 0.0100 M EDTA using xylenol orange as indicator. Stock solutions of 100 mM oxalic and diglycolic acids were prepared. These solutions were diluted and passed through a membrane filter of 0.45 μm pore size (Gelman Science, Tokyo) and degassed by sonification with a Branson Ultrasonic cleaner (Yamato, Tokyo), prior to use. All Pyrex glassware and polyethylene bottles were washed sequentially with a detergent solution, soaked in 10% nitric acid for at least 24 h, and washed three times with pure water.

2.4. The preparation of metal-free sea water

Metal-free sea water was prepared as in Pai's procedure [14] with a slight modification. The bulk of



Scheme 2. Preparation of the stationary phases of chelation ion chromatography.

freshly collected coastal sea water is stored in a polyethylene tank. The sea water (2 l) is filtered with a Whatman GF/C glass fiber filter, then passed through a column containing activated carbon (5 g) to remove organic substances. An aliquot of 10 ml of the iron(III) hydroxide slurry, prepared as in the following procedures, was added and the mixture shaken vigorously, allowed to stand for 48 h until the brownish slurry of iron(III) hydroxide precipitate settled at the bottom. The undisturbed supernatant was siphoned out and led to a Chelex-100 resin column for pretreatment as described below at a flow rate of 5 ml min^{-1}. The first 30 ml of the effluent was discarded and the rest collected and stored in PTFE bottles.

2.5. Iron(III) hydroxide slurry

Iron(III) chloride (0.5 g) and boric acid (0.05 g) were dissolved in pure water (ca. 10 ml), and the pH adjusted to ca. 8 with sodium hydroxide solution to form a brownish slurry.

2.6. Chelex-100 column

5 g of Chelex-100 resin (100–200 mesh, Bio-Rad) were packed into a polyethylene column (30×0.6 cm i.d.). The column was activated by washing sequentially with nitric acid (2 M), pure water; sodium hydroxide (1 M), pure water; magnesium chloride (1 M) and pure water, until the pH of the effluent was pH 8–9. The resin in the column was thus converted into the magnesium form.

3. Results and discussion

Determination of trace elements in complex matrices relies on two processes: the concentration of the analyte trace elements and elimination of the sample matrix that interferes with the determination of the metal ions [2]. Since γ-aminobutyrohydroxamate resin (P13) has a high affinity for the transition metal ions and reacts rapidly with them [11–13], it is suitable for use as the stationary phase of chelation ion chromatography. The chelating resin (P13) was packed in a 50×2 mm concentrator column (Scheme 1) instead of the sample loop. To prevent breakthrough during the loading of a sample onto the resin column and during the flushing of the loaded sample, in order to remove interferences, the capacity of the sorbent and the retention of the analyte in the resin column have to be considered. In this investigation, the precolumn was conditioned with 25 ml of potassium nitrate (0.02 M) until the baseline was stable. Next, the sample was fed straight into the

detector until a stable elevated absorbance signal was obtained. By switching the precolumn in-line, the sample will be enriched on the precolumn and the detector signal will start to record a frontal analysis chromatogram. For the determination of breakthrough capacity, the concentrations of both analyte and interference were considered. For 1 μg ml^{-1} Eu(III), since the times for the breakthrough volume are much longer than 3 h, at flow rates of 1.0 and 3.0 ml min^{-1}, only the tested volume was indicated (≫230 ml for 1.0 ml min^{-1} and >580 ml for 3.0 ml min^{-1}) and no quantitative capacity was determined. For 10 μg ml^{-1} analyte, the breakthrough capacity is 1070 μg at a flow rate of 1.0 ml min^{-1}, while the breakthrough capacity reduces to 540 μg at a flow rate of 3.0 ml min^{-1}. A similar experimental procedure was applied to determine the metal recovery with sample solutions of 100 ng ml^{-1} for each metal ion in pure water and in sea water at a loading flow rate of 2.0 ml min^{-1}. The stripping solution was 0.1 M HCl. The recovery from the precolumn was between 97% and 107% for the metal ions in pure water. Somewhat lower values were obtained for the sea water (between 93% and 105%) (Table 1). The small difference might be due to the hydrophobicity of the polymer matrix, so that apolar compounds in the sample could be loaded on the surface and reduce the capacity of the resin. Another complication is the presence of interfering cations which block the available chelating ion exchange sites and reduce the capacity for ionic analytes.

Table 1
Recovery of lanthanides by γ-aminobutyrohydroxamate resin

Metal ion [a]	Recovery (%)	
	Pure water	Sea water
Sm	105.1	105.3
Eu	104.8	98.2
Gd	105.8	92.9
Tb	96.7	95.6
Dy	106.7	101.1
Ho	97.9	103.7
Er	99.5	93.0

[a] 10 μg added.
Dimension of the concentrator: 50×2 mm i.d. Particle size: 100–230 mesh; Sample solution: 100 ng ml^{-1} for each metal ion; Loading flow rate: 2.0 ml min^{-1}; Stripping solution: HCl (0.1 M); Analytical column: Dionex CS-5; Eluent: oxalic acid (0.03 M, pH 4.5)–diglycolic acid (0.005 M); Detection: PAR (6×10^{-5} M)–ZnEDTA (1×10^{-4} M) in ammonia–ammonium chloride buffer (0.3 M, pH 10) as postcolumn reagent; λ_{max}=495 nm.

Ammonium acetate at pH 5.0–5.5 can be used to elute Na, K, Ca and Mg selectively from a Chelex-100 resin column [2]. In order to investigate the adsorption behavior of the sample matrix on the concentrator, at first 1% magnesium and calcium aqueous solutions were selected as models. Pure water, ammonium acetate, ammonium nitrate, potassium nitrate, sodium nitrate, and boric acid were used as eliminators for the matrix. Since complexation would occur with ammonium salts, resulting in the loss of some analytes and the decrease of absorbance in postcolumn detection, they were abandoned. Other salts, except boric acid, show some potential as matrix eliminators. Pure water seems most convenient and highly promising as the eliminator (Fig. 1) among the tested reagents. In these experiments, oxalic acid was used as the lanthanide eluent, and detection of metal ions was accomplished by postcolumn derivatization using 4(2-pyridylazo)resorcinol (PAR) (6×10^{-5} M) in ammonia–ammonium chloride buffer (0.3 M, pH 10). Large amounts of magnesium were eluted at the retention time of 3 min (Fig. 1); moreover, calcium oxalate would precipitate and block the separator column if a large amount of calcium were not pre-eliminated. When pure water was tested as a possible eliminator, the broad matrix peak disappeared in both 1% Mg and 1% Ca solutions and no precipitation was observed in the separator system (Fig. 1). Unfortunately, this procedure would bring about swelling of the concentrator (P13 resin column). To maintain a constant pressure in the concentrator, the optimum concentration of potassium nitrate (0.02 M, 25 ml) was chosen as the eliminator.

As described above, complexing agents can be used to strip trace metal ions retained by P13 resin concentrator. However, a suitable solution is needed simultaneously to elute trace metal ions retained by the concentrator and separation column. Here, various concentrations of oxalic acid, citric acid, tartaric acid and ethylene diamine were tested. The efficiency of the complexing agent modified with various concentrations of inorganic salts, such as NaCl, $NaNO_3$ and KNO_3 or organic solvents, such as methanol, acetonitrile or acetone were studied. Separations of lanthanides using (*N*-methyl P13)

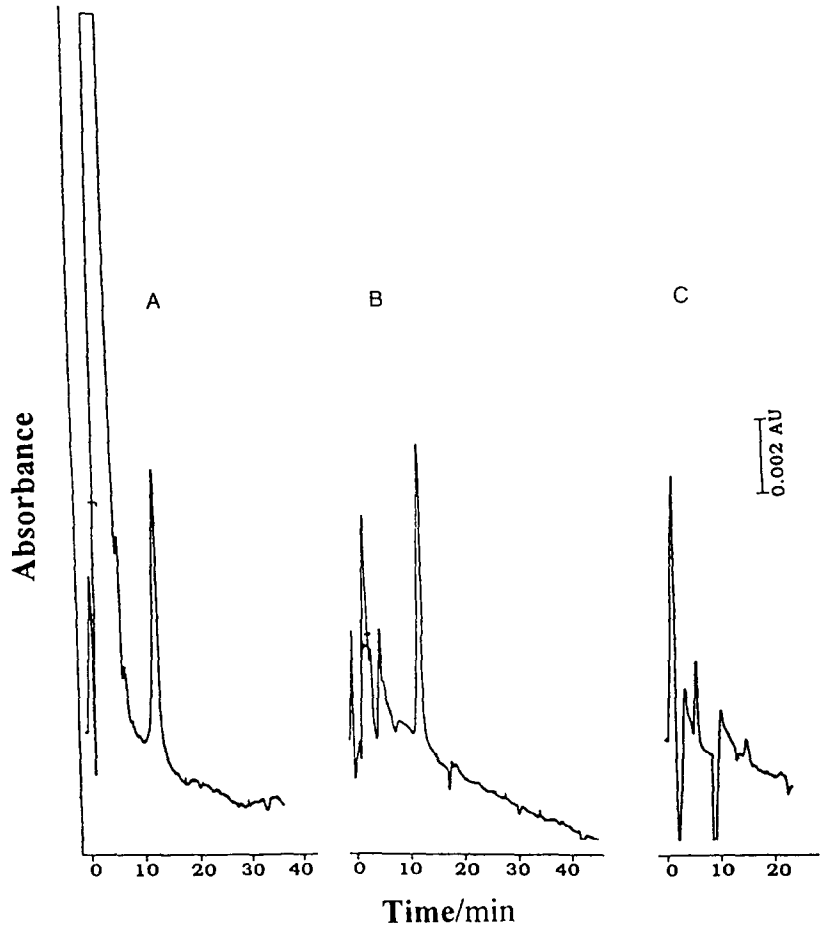


Fig. 1. Chromatogram showing the selectivity of the concentrator. Concentrator: P13 (10×4 mm i.d.); Condition solution: ammonium acetate (0.001 M, 25 ml); Analytical column: Dionex CS-5 (250×4.6 mm i.d.); Eluent: oxalic acid (0.03 M, pH 2.6); Detection: PAR (6×10^{-5} M) in ammonia–ammonium chloride buffer (0.2 M, pH 10); λ_{max}=495 nm. (A) 1% Mg solution (25 ml); (B) 1% Mg solution (25 ml), eliminated with H_2O (50 ml); (C) 1% Ca solution (25 ml), eliminated with H_2O (50 ml).

stationary phases by isocratic and gradient elution were possible (Fig. 2). The elution order is Er<Eu<Sm<La, which can be explained on the basis of their hydration energy. For the lanthanide contraction, the larger the atomic number, the greater the hydration energy, thus making it more difficult to chelate with the resin stationary phase. The chromatograms in Fig. 3 show the separation of metal ions in pure and sea waters under optimum conditions. No significant difference was observed between separations; however, a lower peak height was obtained. The results (Fig. 3B) show that the (*N*-methyl P13) separator column exhibits highly selective lanthanide separations without any noticeable matrix effect, although some drifting phenomena are indicated in the chromatogram.

Separations of rare earth mixture on the Dionex CS-5 stationary phase using gradient elution with oxalic acid (0.06 M, pH 4.5) to oxalic acid (0.02 M)–diglycolic acid (DGA) (0.01 M) in 25 min were also studied. With this eluent, the elution order is La<Ce<Pr<Nd<Eu<Sm<Gd<Tb<Dy<Ho<Er, which is not the same as that for the separation by the (*N*-methyl P13) column. The use of stronger complexing agents, such as oxalate, might result in the formation of anionic lanthanide complexes. Under

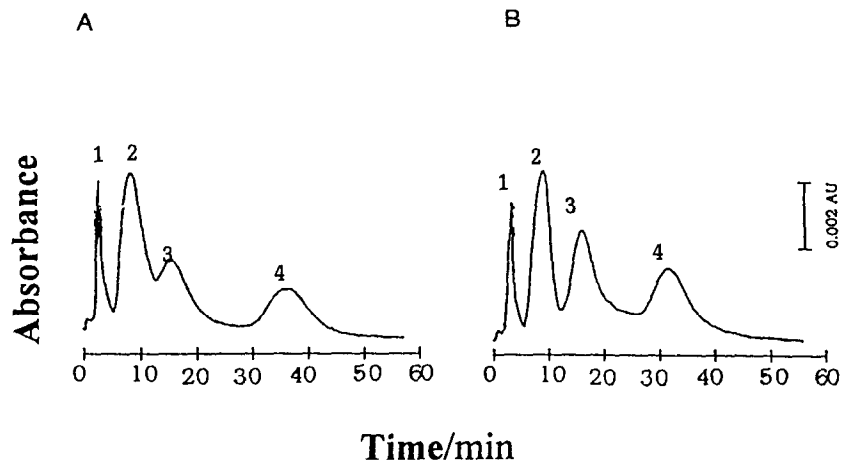


Fig. 2. Separation of selected lanthanides by isocratic and gradient elution. Sample solution: Er(III) (0.89 μg ml^{-1}) – Eu(III) (0.74 μg ml^{-1}) – Sm(III) (0.84 μg ml^{-1}) – La(III) (0.70 μg ml^{-1}); Sample loop: 50 μl; Analytical column: *N*-methyl P13; Mobile phase: (A) isocratic elution with oxalic acid (4×10^{-3} M, pH 4.0); (B) gradient elution.

Time/min	0	3	5	10	15
Condition ($\times10^{-3}$ M)	3	4	3	5	6

Detection: PAR (6×10^{-5} M) in ammonia–ammonium chloride buffer (0.2 M, pH 10); flow rate: 0.5 ml min^{-1}; λ_{max}=520 nm; Peak identification: (1) Er(III); (2) Eu(III); (3) Sm(III); (4) La(III).

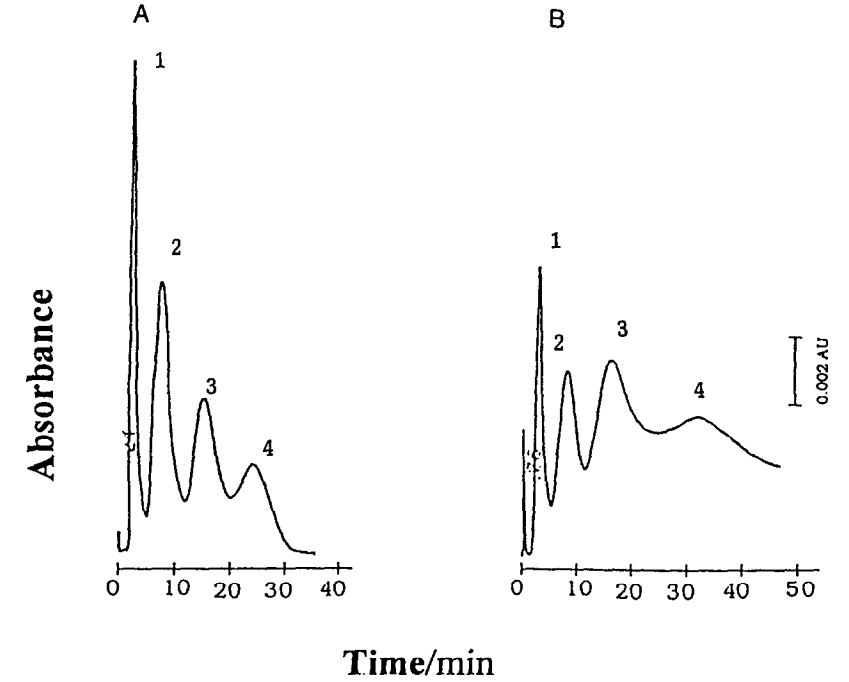


Fig. 3. Comparison of the separations of lanthanides in pure water and sea water. Conditions as Fig. 2(B). except the flow rate of PAR is 0.7 ml min^{-1}; (A) in pure water; (B) in sea water. Peak identification: 1. Er(III); 2. Eu(III); 3. Sm(III); 4. La(III).

these conditions, the lanthanide series may be separated by anion exchange. Because the strongest complexes are the most negatively charged, the elution order is reversed from that of cation exchange separations [15].

A highly selective fluorometric method for the determination of lanthanides has been based on 8-hydroxyquinoline-5-sulfonic acid (HQS) [16,17]. This detection system revealed noise and weak fluorescence intensity which might be due to the stronger complexing capability of the mobile phase, thus competing with HQS. Hence, in this work the lanthanides were detected by measuring the absorbance using PAR (6×10^{-5} M) as the postcolumn reagent. With the addition of Zn-EDTA (1×10^{-4} M) to PAR (6×10^{-5} M), an improvement in detection limit (to 10^{-5} M) for the lanthanides was obtained. The displacement reactions are quite rapid. Under the proposed detection system, however, the baseline exhibits serious drifting and poor resolution of La, Ce and Pr. To solve the drifting phenomena isocratic elution was used. But the resolution did not improve, even when Nd was coeluted with La, Ce and Pr

Table 2
Retention characteristics of the lanthanides

Element	Capacity factor		
	Gradient[a]	Isocratic	
		pure water	sea water
La(III)	2.5	4.5	5.6
Ce(III)	2.5	4.5	5.6
Pr(III)	2.5	4.5	5.6
Nd(III)	4.5	4.5	5.6
Eu(III)	8.5	6.5	7.0
Sm(III)	10.5	8.5	9.0
Gd(III)	12.5	9.5	10.3
Tb(III)	15.5	14.0	15.0
Dy(III)	18.0	16.5	19.0
Ho(III)	19.5	18.5	21.0
Er(III)	23.5	19.5	22.3

Gradient: Sample solution: mixture of La(III), Ce(III), Pr(III), Nd(III), Sm(III), Eu(III), Gd(III), Tb(III), Dy(III), Ho(III) and Er(III) in pure water, 5 μg ml^{-1} each; Sample loop: 50 μl; Analytical column: Dionex CS-5 (250×4.6 mm i.d.); Eluent: oxalic acid (0.06 M, pH 4.5) to oxalic acid (0.02 M)–diglycolic acid (0.01 M) in 25 min.; [a] With PAR(6×10^{-5} M)–ZnEDTA (1×10^{-4} M) in ammonia-ammonium chloride buffer (0.3 M, pH 10) as postcolumn reagent; λ_{max}=495 nm.
Isocratic: Concentrator: γ-aminobutyrohydroxamate resin (50×2 mm i.d.); Condition solution: KNO_3 (0.02 M, 25 ml); Sample solution: 25 ml mixture of La(III), Ce(III), Pr(III), Nd(III), Sm(III), Eu(III), Gd(III), Tb(III), Dy(III), Ho(III) and Er(III) (10 ng ml^{-1} each); Loading flow rate: 2.0 ml min^{-1}; Eliminator: KNO_3 (0.02 M, 25 ml); Analytical column: Dionex CS-5 (250×4.6 mm i.d.); Eluent: oxalic acid (0.03 M, pH 4.5)–diglycolic acid (0.005 M); Detection: PAR (6×10^{-5} M)–ZnEDTA (1×10^{-4} M) in ammonia-ammonium chloride buffer (0.3 M, pH 10); λ_{max}=495 nm.

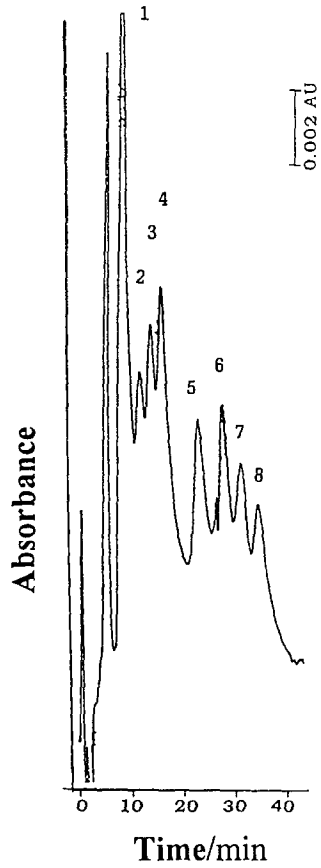


Fig. 4. On-line preconcentration and separation of lanthanides in sea water. Concentrator: γ-aminobutyrohydroxamate resin (50×2 mm I.D.); Condition solution: KNO_3 (0.02 M, 25 ml); Sample solution: 25 ml mixture of La(III), Ce(III), Pr(III), Nd(III), Sm(III), Eu(III), Gd(III), Tb(III), Dy(III), Ho(III) and Er(III) (10 ng ml^{-1} for each); Loading flow rate: 2.0 ml min^{-1}; Eliminator: KNO_3 (0.02 M, 25 ml); Analytical column: Dionex CS-5 (250×4.6 mm I.D.); Eluent: oxalic acid (0.03 M, pH 4.5)–diglycolic acid (0.005 M); Detection: PAR (6×10^{-5} M)–ZnEDTA (1×10^{-4} M) in ammonia–ammonium chloride buffer (0.3 M, pH 10) as post-column reagent; λ_{max}=495 nm; Peak identification: 1. La(III), Ce(III), Pr(III) and Nd(III); 2. Sm(III); 3. Eu(III); 4. Gd(III); 5. Tb(III); 6. Dy(III); 7. Ho(III); 8. Er(III).

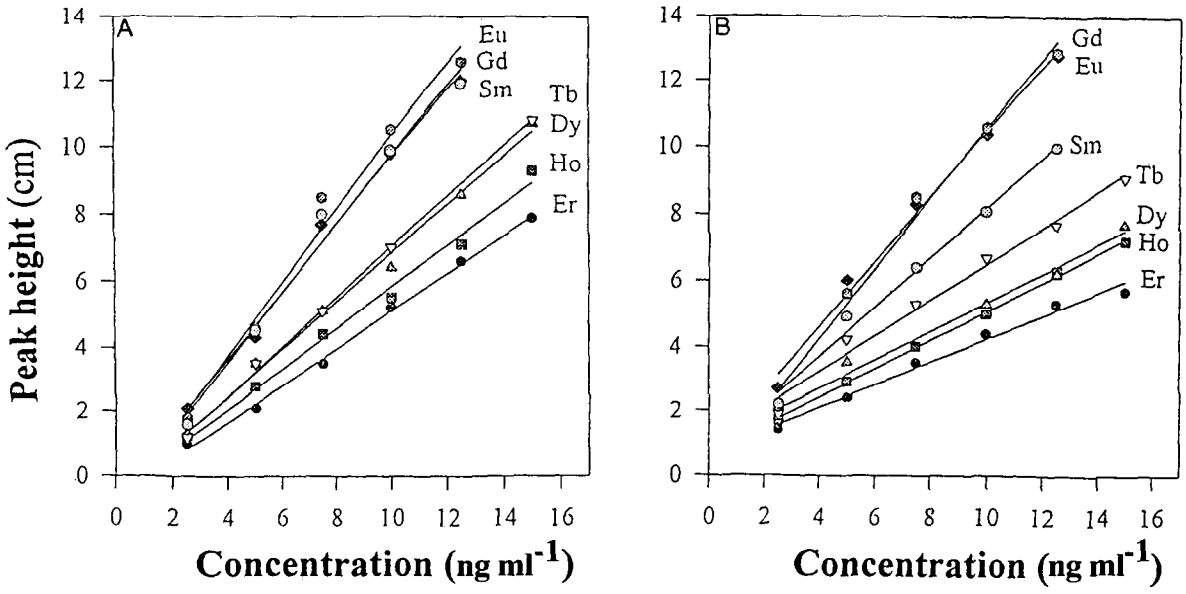


Fig. 5. Calibration graphs for the determination of lanthanides. Conditions as in Fig. 4. (a) in pure water; (b) in sea water.

(Table 2). Improvements in resolution may be achieved after further study. The capacity factor in Table 2 is defined conventionally; $(t_r-t_m)/t_m$, where t_m is the time for the unretained mobile phase to travel through the column and t_r is that for the analytes.

With oxalic acid (0.03 M, pH 4.5)–diglycolic acid (0.005 M) as eluent, P13 resin concentrator and PAR-ZnEDTA as the postcolumn reagent, a just passable separations of 10 ng l^{-1} each of Eu(III), Sm(III), Gd(III), Tb(III), Dy(III), Ho(III) and Er(III) in sea water was obtained, but not of La(III), Ce(III), Pr(III) and Nd(III) (Fig. 4). The analytical performance was also evaluated. Although the linear response range is small, within the concentration range studied (2–16 ng ml^{-1}), good linear correlation between peak heights and concentrations was obtained for each species (Fig. 5). In this study, calibration was obtained by spiking standard metal ions in the case of the metal-free sea water matrix. A small variation in slope of the calibration lines was seen in pure water and sea water matrices (Fig. 5). The results indicate that the prepared resin is highly promising as a preconcentrator. The detection limits for the lanthanides defined on the basis of three times the standard deviation of the response for the lowest concentration (n=5) in the chromatogram are 2.5 ng ml^{-1}, obtained by concentrating 25 ml of sample onto the analytical system both from pure water and sea water matrices.

4. Conclusion

With a preconcentrator, the proposed system is able to determine trace metal species at the ng ml^{-1} level. The present scheme for the determination of lanthanides in sea water is simple and rapid. The automated on-line operation requires only 25 ml of the sample. Sea water was selected as a general matrix for the development of a chelation ion chromatography method since it contains trace quantities of metal ions in the presence of high concentrations of alkali and alkaline earth elements. With *N*-methyl P13 as separator or P13 as concentrator, there is a great potential for the separation of complex matrix samples. Moreover, a higher separation efficiency and a wider field application would be expected for smaller resin particles or even more selective detection methods. The proposed method for the determination of lanthanides in sea water would be useful for determination of these metal ions

in biological samples or for their recovery from complex matrix samples.

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